

The onset of the Permo-Carboniferous glaciation: reconciling global stratigraphic evidence with biogenic apatite $\delta^{18}\text{O}$ records in the late Viséan.

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Abbreviated title: Late Viséan cooling marks onset of LPIA phase.

Abstract. The $\delta^{18}\text{O}$ values of phosphatic microfossils recovered from NW Ireland are used to determine the timing and magnitude of cooling associated with the onset of the Carboniferous glaciation. Microfossil fish $\delta^{18}\text{O}_{\text{apatite}}$ demonstrates a +2.4‰ (V-SMOW) shift, which, once corrected for $\delta^{18}\text{O}_{\text{seawater}}$ changes due to evolving ice-volumes, equates to an approximate 4.5 °C reduction in equatorial sea-surface-temperature (SST) between the basal Asbian and the mid-Brigantian (late Viséan). Both conodont and microfossil fish $\delta^{18}\text{O}_{\text{apatite}}$ indicate stabilisation of an “Ice-House” climate

24 during the Brigantian and into the Serpukhovian. Substantial upper Viséan cooling identified herein is
25 in good agreement with global glacioeustatic records.

26

27 **Supplementary material:** Further information on the palaeogeography, lithostratigraphy and
28 palaeoenvironments of the sections examined as well as the sample composition and analytical
29 methodology of oxygen isotope analyses is available at www.geolsoc.org.uk/SUP00000.

30 The Permo-Carboniferous glaciation (late Palaeozoic ice age [LPIA]) is recognised as one of the most
31 significant in Earth's history and has also been identified as an important analogue for modern climate
32 (Montañez, *et al.* 2007). Despite this, considerable disagreement remains, particularly concerning the
33 timing of the main phase of glaciation. Earlier studies proposed a relatively simplistic protracted
34 glaciation throughout much of the Carboniferous and Permian (e.g. Veevers & Powell 1987). More
35 recently, discrete glacial and temperate episodes have been recognised superimposed on larger-scale
36 climate trends (e.g. Fielding, *et al.* 2008).

37 Glaciogenic deposits and erosional surfaces provide, essentially, the only direct evidence for past
38 glaciations. However, significant difficulties are associated with their use in this respect, since they are
39 often undateable, extensively reworked or even completely eroded during subsequent glacial advances
40 (Frank, *et al.* 2008). For this reason, distal proxies have become popular for reconstructing ancient
41 "Ice-house" phases. By definition though, proxies only provide a substitute record and all of the
42 controlling factors of the 'system' must be well-understood before palaeoclimatic interpretations are
43 attempted.

44 The proliferation in the use of stable isotopes, particularly those of oxygen, to construct high-resolution
45 records of palaeoenvironmental shifts has demonstrated the potential for well-understood proxy
46 systems. The bulk of published work has focussed on utilising carbonates as isotopic media; however,
47 as ancient carbonates are susceptible to diagenetic alteration, screening of older samples is vital.
48 Although methods have been developed to assist in selecting only well-preserved carbonate samples for
49 analysis (e.g. Mii, *et al.* 2001), the potential for carbonate alteration has led to an expansion in the use
50 of biogenic phosphate as an alternative isotopic reservoir. The relative high-fidelity of the isotopic
51 signal preserved by biogenic phosphate has been demonstrated through a number of histological and
52 geochemical studies (e.g. Trotter, *et al.* 2007, Joachimski, *et al.* 2009). Isotopic analyses of Palaeozoic
53 phosphates are predominantly undertaken on the bio-apatite of ichthyolith (fish microfossil) and

conodont elements. The latter represent the only mineralised parts of an extinct group of small, pelagic or nekto-benthic, primitive, jawless marine vertebrates (Aldridge, *et al.* 1993). Importantly, conodont elements have an outer layer of hyaline tissue, which, like ichthyolith enameloid, is densely crystalline and ideal for preserving primary $\delta^{18}\text{O}$ signatures.

During the Early Carboniferous, Ireland was located just south of the palaeoequator and marine sediments accumulated across a series of shallow platforms and basins during a transgressive phase spanning the suspected onset of the LPIA. Conodonts and ichthyoliths recovered from five related, late Viséan to Serpukhovian, thermally mature (conodont colour alteration index [CAI] of *c.* 3.5), sedimentary sequences, were isotopically analysed. The predominantly shallow marine succession examined in NW Ireland represents a unique opportunity to examine the onset of the LPIA due to (i) the relatively expanded temporal record preserved, (ii) commonly occurring ichthyolith and conodont elements *and* (iii) good biostratigraphical control.

Results. The results of oxygen isotope analyses on conodont ($n = 12$) and ichthyolith ($n = 36$) apatite samples are presented in Fig. 1. $\delta^{18}\text{O}_{\text{ichthyolith}}$ values show an increasing trend from the basal Asbian to the Brigantian, ranging from 15.1 to 19.1‰ (V-SMOW). Conodont and ichthyolith $\delta^{18}\text{O}$ values are relatively stable throughout the Brigantian with conodonts demonstrating a slight decrease from the mid-Brigantian into the Serpukhovian (21.1‰ to 20.2‰ V-SMOW). $\delta^{18}\text{O}_{\text{ichthyolith}}$ values were found to be relatively consistently depleted in ^{18}O by an average of $2.0\text{‰} \pm 0.6\text{‰}$ (1σ , $n = 5$) in comparison to conodonts recovered from either the same sample or laterally equivalent stratigraphic levels (Fig. 1). Where multiple samples were analysed from the same level, no statistically significant, consistent $\delta^{18}\text{O}$ differences were observed which indicate palaeoecological effects.

77 **Discrepancy between conodont and ichthyolith $\delta^{18}\text{O}$.** A “vital-effect” is discounted as an
78 explanation for the offset between the two phosphatic media analysed since no evidence exists for the
79 fundamental physiological difference between fish and conodonts which would be required to explain
80 the essentially stable discrepancy across the range of taxa analysed. Additionally, an offset of a greater
81 magnitude has been identified between similar fish and conodont taxa in coeval but thermally more
82 mature strata elsewhere in Ireland (Barham 2010).

83 Neither can palaeoecology explain the 2‰ difference between conodonts and ichthyoliths, since
84 significant overlap is expected in the habitats of the conodonts and fish analysed. The average $\delta^{18}\text{O}$
85 offset would hypothetically translate either into a habitat palaeotemperature difference of 8 to 9 °C
86 (Pucéat, *et al.* 2010) or a significant difference in salinity. It is difficult to conceive such large,
87 consistent, primary $\delta^{18}\text{O}_{\text{seawater}}$ differences either vertically within the tropical water column or laterally
88 across the predominantly shallow platform studied.

89 Alternatively, it has been suggested that conodont elements, due to their dense apatite structure (like
90 enamel), more accurately preserve primary environmental signals, whilst the more intrinsically porous
91 Palaeozoic ichthyoliths are prone to isotopic depletion via interaction with relatively ^{18}O -depleted
92 diagenetic fluids (Barham 2010, Žigaite, *et al.* 2010). However, the mechanism of a relatively stable
93 diagenetic O-exchange induced isotopic offset is difficult to conceive and inorganic O-exchange with
94 the PO_4^{3-} group occurs very slowly (Blake, *et al.* 1997). The deposition of ^{18}O -depleted apatite within
95 the primary pore spaces of ichthyoliths could also account for the observed isotopic offset, although no
96 significant or consistent microscopic evidence for this was observed. $\delta^{18}\text{O}_{\text{ichthyolith}}$ values were
97 interpreted to be less faithful than those of conodonts due to the more plausible palaeotemperatures
98 derived from $\delta^{18}\text{O}_{\text{conodont}}$ values. Using the equation of Pucéat *et al.* (2010), temperatures calculated on
99 $\delta^{18}\text{O}_{\text{ichthyolith}}$ in part exceeded 50 °C, whilst $\delta^{18}\text{O}_{\text{conodont}}$ values translate into more viable
100 palaeotemperatures of 30 to 35 °C, considering the palaeogeographic setting of Ireland and

101 uncertainties in estimating the absolute oxygen isotopic composition of Mississippian seawater (Puc  at,
102 *et al.* 2010). In addition, $\delta^{18}\text{O}_{\text{conodont}}$ values are correlatable: average late Vis  an $\delta^{18}\text{O}_{\text{conodont}}$ values of
103 20.7   from NW Ireland are compatible with coeval average values of 21   reported by Buggisch *et*
104 *al.* (2008) from northern Spain. Furthermore, conodont elements consistently appeared lustrous whilst
105 ichthyoliths, in contrast, commonly displayed coarsened, partially recrystallised basal tissue indicating
106 a different preservation of both phosphatic microfossils.

107 Although $\delta^{18}\text{O}_{\text{ichthyolith}}$ values appear to have been affected by diagenesis, the offset from (primary)
108 $\delta^{18}\text{O}_{\text{conodont}}$ values is relatively uniform, which is consistent with the comparative work of Barham
109 (2010) and   igaite *et al.* (2010). We tentatively suggest that in order to compensate for this diagenetic
110 effect, $\delta^{18}\text{O}_{\text{ichthyolith}}$ values may be corrected through comparison with temporally and spatially coeval
111 conodonts. $\delta^{18}\text{O}_{\text{ichthyolith}}$ values in this study may therefore be translated by the average offset of +2.0  
112 to best estimate their original values and reconstruct a corrected-ichthyolith-conodont $\delta^{18}\text{O}$ dataset.

113 Despite probable diagenetic overprinting of ichthyolith oxygen isotope values, the positive trend in the
114 Asbian may be interpreted as primary and representative of palaeoclimatic conditions since: (i) the
115 steady increase in $\delta^{18}\text{O}_{\text{ichthyolith}}$ is statistically unlikely if the data represent a “*random walk*”, (ii)
116 thermal gradients are insufficient to explain the magnitude of the observed positive trend.

117 Hypothetically, the $\delta^{18}\text{O}$ increase through the Asbian could be the sole result of complete overprinting
118 at a relatively low temperature (40 to 50   C) and a plausible thermal gradient of *c.* 30   C/km. However,
119 complete overprinting of $\delta^{18}\text{O}_{\text{ichthyolith}}$ values is improbable at these relatively low temperatures without
120 enzyme mediation (Blake, *et al.* 1997). It might also be expected that complete overprinting would
121 result in lower $\delta^{18}\text{O}$ values reflecting the peak temperatures experienced during diagenesis (*c.* 200  C
122 according to regional CAI values). Furthermore, partial thermal gradient overprinting would require a
123 hypothetical gradient much steeper than that developed in the region (Corcoran & Clayton 2001).

124 Finally, $\delta^{18}\text{O}_{\text{ichthyolith}}$ values remain stable through Brigantian strata despite similar thermal gradients,

125 (iii) $\delta^{18}\text{O}_{\text{ichthyolith}}$ values from coeval but geographically separate strata are compatible, *and* (iv) as
126 discussed below, the isotopic evolution identified is palaeoenvironmentally plausible.

127

128 **Palaeotemperature and palaeoclimate interpretations.** Regardless of whether absolute $\delta^{18}\text{O}_{\text{ichthyolith}}$
129 values can be corrected for an offset, the overall increasing $\delta^{18}\text{O}$ trend can be interpreted independently
130 in terms of palaeoenvironmental change since the trend retains a primary signal. The positive oxygen
131 isotope shift should, therefore, represent changes in $\delta^{18}\text{O}_{\text{seawater}}$ associated with the evolution of the
132 depositional environment and the expansion of continental ice-masses, as well as cooling of SST.
133 Changes in local salinity may have affected $\delta^{18}\text{O}_{\text{seawater}}$ (the interval of highest $\delta^{18}\text{O}_{\text{apatite}}$ values broadly
134 coincides with the most restricted conditions). However, the majority of the positive $\delta^{18}\text{O}$ trend is
135 correlated with platform carbonates with no substantial salinity changes being evident from the
136 sedimentology or faunal composition. Furthermore, $\delta^{18}\text{O}_{\text{apatite}}$ values are relatively stable from the
137 Brigantian onwards despite a large range of lithologies (and environments) being sampled in an often
138 rapidly fluctuating sequence *and* average $\delta^{18}\text{O}_{\text{conodont}}$ values from the most restricted depositional
139 interval analysed, are essentially identical to those reported from Spain.

140 Determining the magnitude of SST cooling requires knowledge of the change in $\delta^{18}\text{O}_{\text{seawater}}$ as a
141 consequence of the growth of isotopically light ice-masses (Fig. 2). It is proposed that the climate of the
142 early- to mid-Viséan may have been transitional to icehouse (Shi & Waterhouse 2010), i.e. broadly
143 akin to the present ($\delta^{18}\text{O}_{\text{seawater}} = 0\text{‰}$ V-SMOW) or less glaciated ($\delta^{18}\text{O}_{\text{seawater}} = -0.5\text{‰}$ V-SMOW). By
144 Serpukhovian times, ice-sheets are interpreted to have been comparable in extent to the Last Glacial
145 Maximum (LGM; González-Bonorino & Eyles 1995), with $\delta^{18}\text{O}_{\text{seawater}}$ as high as 1.2‰ (Fairbanks
146 1989). Since this dataset does not continue far into the Serpukhovian, a $\delta^{18}\text{O}_{\text{seawater}}$ value of 1‰ (V-
147 SMOW) is assumed for the peak glacial conditions identified here. This implies that the formation of
148 ice sheets in Gondwana may have accounted for *c.* 1.5‰ of the total 2.5‰ increase documented in

$\delta^{18}\text{O}_{\text{apatite}}$. The remaining change of 1‰ is interpreted to reflect approximately 4 to 5 °C cooling, comparable to tropical SST change during the LGM (Beck, *et al.* 1997). It is arguable that this estimate represents a cooling minimum since the marine lithologies sampled may not capture glacial maxima. This may also explain why no obvious ice-sheet coupled isotopic fluctuation is recorded.

Comparison of results to other studies. Fig. 3 compares the results of this work with a number of significant studies on the LPIA. The Viséan-Serpukhovian boundary has been identified as marking the start of significant climate deterioration using sedimentological data (Fig. 1 of Fielding, *et al.* 2008) and oxygen isotope data (Mii, *et al.* 2001, Buggisch, *et al.* 2008). However, late Viséan cooling has recently been suggested based on $\delta^{18}\text{O}$ values of brachiopod shells from Spain (Armendáriz, *et al.* 2008). More significantly, sedimentary cyclicity and eustatic fluctuations with Milankovitch periodicities have been reported from Asbian and Brigantian strata: (i) in the northwest Irish sections examined (Schwarzacher 1989, Barham 2010), (ii) elsewhere in Ireland and Britain (Wright & Vanstone 2001), (iii) in equivalent Canadian sequences (Giles 2009), (iv) in the stratigraphically significant Arrow Canyon Section in Nevada, America (Bishop, *et al.* 2009), and (v) elsewhere globally (Rygel, *et al.* 2008 and references therein). The compatibility of this collective body of work strongly indicates an initiation of cooling before the Viséan-Serpukhovian transition (Fig. 3).

The reasons for the disagreement between supposedly globally representative oxygen isotope records are not entirely clear at present. Despite the usefulness illustrated by isolated studies of low-magnesium brachiopod calcite, the dataset compiled by Frank *et al.* (2008) demonstrates (i) the significant spread of $\delta^{18}\text{O}_{\text{carbonate}}$ data relative to $\delta^{18}\text{O}_{\text{apatite}}$, and (ii) the incoherence of the trendline, which bears little or no correlation with any other indicators of the LPIA, produced by combining the results of studies from different palaeogeographic areas.

172 The palaeogeographic spread of samples is a particularly pertinent consideration since the majority of
173 oxygen isotope studies are based on sequences deposited in epeiric seas which, due to their somewhat
174 restricted nature are more susceptible to local palaeoenvironmental-induced heterogeneities in
175 $\delta^{18}\text{O}_{\text{seawater}}$ (Brand, *et al.* 2009). It may be inaccurate to compare the absolute values of coeval oxygen
176 isotope analyses from different continental seas since they likely had different $\delta^{18}\text{O}_{\text{seawater}}$ properties.
177 The sedimentary strata examined in this study were all deposited from the same regional watermass and
178 where stratigraphic overlap exists, $\delta^{18}\text{O}_{\text{apatite}}$ values are found to be very consistent (Fig. 1). In any case,
179 only the $\delta^{18}\text{O}$ trend in this study is given particular credence.

180 Discrepancies between studies utilising different fossil hardparts may be explained by currently
181 unrecognised physiological effects or differential diagenetic susceptibilities. More generally,
182 inadequate or different bio- and lithostratigraphic constraints and incomplete stratigraphic resolution of
183 the extremely dynamic climate system may explain conflicting results. In the case of the
184 comprehensive $\delta^{18}\text{O}_{\text{apatite}}$ dataset of Buggisch *et al.* (2008), an inability to correlate late Viséan to early
185 Serpukhovian isotopic curves may be explained by (i) the variable diagenetic conditions experienced
186 by the Cantabrian Sections (CAI 1.5 to 7), *and/or* (ii) difficulties associated with precisely plotting data
187 from several condensed Cantabrian Sections. This paper is based on over 2.5 times as much data,
188 within a stratigraphy which is approximately 20 times more expanded, than that of Buggisch *et al.*
189 (2008), making for a more resolved picture of climate dynamics during this interval. However, the
190 discrepancies between the various studies clearly indicate the need for more research during this critical
191 climatic interval.

192

193 **Implications for the onset of the LPIA.** The correlation between increasing $\delta^{18}\text{O}_{\text{apatite}}$ values and the
194 global litho- and sequence-stratigraphic records of glacioeustatic fluctuations represents the first
195 integration of proxies supporting a late Viséan onset for a significant phase of the LPIA. It is proposed

196 that rapid climate deterioration began at the base of the Asbian and by Brigantian times the magnitude
197 of glaciation was comparable to the LGM. An “Ice-House” climate state was maintained from mid-
198 Brigantian times and, although limited to a single datum, no rapid degradation of climate apparently
199 occurred at the Viséan-Serpukhovian boundary.

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281
282

283 **Fig. 1.** $\delta^{18}\text{O}$ data of conodont and ichthyolith apatite. Columns from the left illustrate western European
284 Stages, British Substages and conodont biozones respectively. Question marks at the Asbian-Brigantian
285 boundary represent a current disagreement in the local biostratigraphy (Sevastopulo & Wyse Jackson
286 2009, and references therein).

287

288 **Fig. 2.** Illustration of how SST interpretations may vary depending on assumptions of initial (Init.) and
289 temporal evolution (Δ) of the isotopic composition of seawater. Note that the absolute temperature
290 scale is based on the (likely) erroneous assumption that $\delta^{18}\text{O}_{\text{seawater}}$ has not changed through time. The
291 trendlines plotted were produced by combining the conodont and ichthyolith datasets after a +2‰
292 translation of $\delta^{18}\text{O}_{\text{ichthyolith}}$ values.

293

294 **Fig. 3.** Comparison of climate proxies used to reconstruct the onset of the LPIA. Background shading
295 indicates periods suggested to be glaciated by that particular study. Black arrows mark the proposed
296 onset of significant cooling at the base of the Asbian. All data are plotted relative to the timescale of
297 Davydov *et al.* (2004).





